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OCTA(ISOPROPOXY)DIMOLYBDENUM AND BIS(NITROSYL)HEXA(ISOPROPOXY)D--ETC(U)

NOV 77 M H CHISHOLM, F A COTTON, M W EXTINE

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Octa(isopropoxy)dimolybdenum and Bis(nitrosyl)hexa
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Containing Molybdenum Atoms with Fourteen
Valence Shell Electronic Configurations.

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between the two structures are (i) the Mo-to-Mo distances which are 3.335(2) and 2.525(1) Å, for I and II, respectively, and (ii) the angles of the $\text{Mo}_2(\mu\text{-O})_2$ moiety. These differences are rationalized in terms of simple ligand field considerations. In compound I, which contains molybdenum in a formal oxidation state of +2, there are four electrons in d_{xz} , d_{yz} atomic orbitals which are extensively involved in back bonding to the NO^+ ligand. In compound II, which contains molybdenum in a formal oxidation state of +4, there are only two electrons in the d_{xz} , d_{yz} atomic orbitals. These electrons then form a Mo-Mo double bond; this accounts for the short Mo-to-Mo bond distance and the diamagnetic nature of compound II.

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Octa(isopropoxy)dimolybdenum and Bis(nitrosyl)hexa(isopropoxy)dimolybdenum.

Structure and Bonding in Compounds Containing Molybdenum Atoms with
Fourteen Valence Shell Electronic Configurations.

Sir:

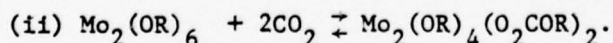
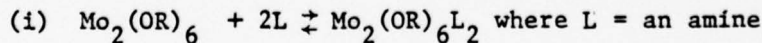
The occurrence of compounds containing metal-to-metal bonds of order 4,3,2 and 1 is now well recognized.¹ As yet, however, the systematic manner in which C-C bond order may be changed in organic chemistry has no parallel in transition metal chemistry. The products of addition/elimination reactions involving dinuclear compounds appear to be unpredictable, although a subsequent rationale may usually be advanced with judicious hindsight. We report here the structural characterization of two closely related and yet contrasting compounds, namely, $\text{Mo}_2(\text{OPr}^i)_6(\text{NO})_2$, I, and $\text{Mo}_2(\text{OPr}^i)_8$, II. Each one may be considered formally as an addition product of $\text{Mo}_2(\text{OPr}^i)_6$, a compound containing a metal-to-metal triple bond.² Compound I is indeed directly formed by the addition of NO (2 equiv) to $\text{Mo}_2(\text{OPr}^i)_6$.³ $\text{Mo}_2(\text{OPr}^i)_8$ is best prepared from the reaction between $\text{Mo}(\text{NMe}_2)_4$ and Pr^iOH .⁴ The addition of 2 NO causes cleavage of the M-M triple bond, while addition of $2\text{Pr}^i\text{O}\cdot$ (a hypothetical reaction) transforms an M-M triple bond to an M-M double bond.⁵

Figure 1 shows the essential features of the coordination geometry in each molecule. Pertinent bond distances and angles are given in Tables I and II. Each compound has rigorous inversion symmetry and virtual C_{2v} symmetry and in each there is essentially trigonal bipyramidal coordination about each molybdenum atom. The asymmetry of the central planar $\text{Mo}_2(\text{u-o})_2$ moiety is most pronounced in I due to the high trans-influence⁶ of the nitrosyl ligands which occupy the equatorial axial positions. The most striking differences between the two structures

are (i) the Mo-to-Mo distances which are 3.335(2) and 2.525(1) \AA , for I and II, respectively, and (ii) the angles of the $\text{Mo}_2(\mu\text{-O})_2$ moiety.

With a Mo-Mo distance of 3.335(2) \AA it may be safely assumed that no significant metal-to-metal bond exists in I, while in II, a Mo-Mo distance of 2.525 \AA is suggestive of a metal-to-metal double bond. This conclusion is supported by the following considerations of bonding. A trigonal bipyramidal field splits the metal d orbitals into three sets $e'(d_{x^2-y^2}, d_{xy})$, $e''(d_{xz}, d_{yz})$ and $a'(d_z)$ with the d_{xz} , d_{yz} degenerate pair lying lowest in energy. In I, each Mo atom may be assumed, formally, to have four 4d electrons after the formation of σ bonds to each of the five ligands, provided we also use the conventional though purely formal description of the linear Mo-N-O moiety as $\text{Mo}^-(\text{NO}^+)$. These four electrons should then fill up the $e''(d_{xz}, d_{yz})$ orbitals, where they can participate very effectively in backbonding to the NO, thus explaining the very low (1632 cm^{-1}) value of ν_{NO} . In compound II, where the formal oxidation number of Mo is +2, each Mo atom has two 4d electrons. It is possible to envision the formation of a double bond as the result of $d_{xz}-d_{xz}$ and $d_{yz}-d_{yz}$ overlaps. This could be construed as a combination of one π bond and one δ bond, but whether the lower symmetry that actually exists will materially alter such a formal description is problematic. In any event, in both I and II the molybdenum atoms have 14-electron valence shell configurations.

Finally, we note two other reactions of $\text{Mo}_2(\text{OR})_6$ compounds which result in the formation of fourteen valence shell electronic configurations for molybdenum:²



Here the M-M triple bond is retained ($\text{Mo-Mo} = 2.242(2)\text{\AA}$) between molybdenum atoms which are four coordinated.⁷

Acknowledgements

We thank the donors of the Petroleum Research Fund administered by the American Chemical Society, the Office of Naval Research and the National Science Foundation (Grant MPS-73-05016) at Princeton University and the National Science Foundation (Grant No. CHE-75-05509) at Texas A&M University for support of this work.

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1. F. A. Cotton, Chem. Soc. Rev., **4**, 27 (1975).
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3. M. H. Chisholm, F. A. Cotton, M. W. Extine and R. L. Kelly, J. Amer. Chem. Soc., submitted for publication.
4. M. H. Chisholm, W. W. Reichert and P. Thornton, J. Amer. Chem. Soc., submitted for publication.
5. Crystals of I and II were grown from hexane solutions.

no P Data for both compounds were collected at 23°C on a Syntex P $\bar{1}$ auto-diffractometer using monochromatized MoK α radiation ($\lambda = 0.710730\text{\AA}$). Unique data having $0.0^\circ < 2\theta_{\text{MoK}\alpha} < 45.0^\circ$ were collected and data having $I > 3\sigma(I)$ were considered observed and used in structure solution and refinement. Structures were solved and refined using the Enraf-Nonius Structure Determination Package and a PDP 11/45 computer owned by Molecular Structure Corporation, College Station, Texas.

no P Crystal data for $\text{Mo}_2(\text{OPr}^{\text{I}})_3$: $a = 9.902(2)$, $b = 17.867(3)$, $c = 9.725(2)\text{\AA}$, $\beta = 102.89(1)$, $V = 1677.2(9)\text{\AA}^3$, $Z = 2$, space group = $P2_1/n$. The molecule has C_1 symmetry. Refinement of non-hydrogen atoms (1826 obs. and 154 variables) employing anisotropic thermal parameters yielded $R = 0.040$ and $R_w = 0.068$.

no P Crystal data for $\text{Mo}_2(\text{OPr}^{\text{I}})_6(\text{NO})_2$: $a = 10.823(1)$, $b = 15.848(2)$, $c = 9.885(2)\text{\AA}$, $\alpha = 90.21(2)$, $\beta = 115.93(2)$, $\gamma = 82.42(1)^\circ$, $V = 1509.4(4)\text{\AA}^3$, $Z = 4$, space group $P\bar{1}$. There are two unique molecules per asymmetric unit, each possessing C_1 symmetry. Refinement of non-hydrogen atoms (2052 obs. and 181 variables) employing anisotropic thermal parameters for the Mo, O, and N atoms and isotropic thermal parameters for the carbon atoms yielded $R = 0.061$ and $R_w = 0.093$.

- G*
6. T. B. Appleton, H. C. Clark and L. E. Manzer, Coord. Chem. Rev., **10**, 335 (1973).
 7. M. H. Chisholm, F. A. Cotton, M. W. Extine and W. W. Reichert, J. Amer. Chem. Soc., in press.
 8. Alfred P. Sloan Fellow 1976-78.

Table I. Selected Bond Distances (Å) and Angles (Deg) in $\text{Mo}_2(\text{OPr}^1)_8$.^a

Atoms	Distance	Atoms	Angle
Mo-Mo'	2.523(1)	Mo-Mo'-O(4)	105.1(1)
-O(1)	1.958(3)	O(1)-Mo-O(1)'	103.5(1)
-O(1)'	2.111(3)	-O(2)	120.9(1)
-O(2)	1.872(3)	-O(3)	83.5(1)
-O(3)	1.976(3)	-O(4)	120.2(2)
-O(4)	1.884(3)	O(1)'-Mo-O(2)	84.9(1)
		-O(3)	173.1(1)
		-O(4)	81.0(1)
Atoms	Angle		
Mo'-Mo-O(1)	54.45(9)	O(2)-Mo-O(3)	91.2(1)
-O(1)'	49.00(9)	-O(4)	118.9(2)
-O(2)	108.9(1)	O(3)-Mo-O(4)	95.8(2)
-O(3)	137.9(1)		

^aAtoms are labelled as in Figure 1A. Esd's are in parentheses.

Table II. Selected Bond Distances (Å) and Angles (Deg) in $\text{Mo}_2(\text{OPr}^1)_6(\text{NO})_2$.^a

Atoms	Molecule I	Molecule II
	Distance	
Mo(1)-Mo(1)'	3.334(2)	3.337(2)
O(1)	1.951(6)	1.946(6)
O(1)'	2.195(6)	2.194(6)
O(2)	1.850(7)	1.849(8)
O(3)	1.861(6)	1.857(7)
N(1)	1.747(9)	1.761(10)
N(1)-O(4)	1.205(11)	1.184(11)
	Angles	
Mo(1)'-Mo(1)-O(1)	39.1(2)	38.9(2)
O(1)'	34.1(2)	33.8(2)
O(2)	102.8(2)	102.8(3)
O(3)	101.4(2)	102.6(2)
N(1)	137.9(3)	138.0(3)
O(1)-Mo(1)-O(1)'	73.1(3)	72.7(3)
O(2)	119.3(3)	119.1(3)
O(3)	117.3(3)	118.6(3)
N(1)	98.8(4)	99.1(4)
O(1)'-Mo(1)-O(2)	88.4(3)	84.6(3)
O(3)	83.8(3)	84.6(3)
N(1)	171.9(4)	171.9(4)
O(2)-Mo(1)-O(3)	115.1(3)	114.2(3)
N(1)	100.8(4)	100.0(4)
O(3)-Mo(1)-N(1)	99.5(4)	99.4(4)
Mo(1)-N(1)-O(4)	178(1)	177(1)

^aAtoms are labelled as in Figure 1b. Esd's are in parentheses.

Table III. POSITIONAL AND THERMAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS. for Mo₂(OPr^t)₈.

Atom	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo	0.01279(5)	0.06291(3)	0.06040(5)	0.00689(5)	0.00182(2)	0.00756(6)	-0.00019(5)	0.00315(9)	-0.00019(5)
O(1)	0.0269(4)	0.0441(2)	-0.1343(4)	0.0078(4)	0.0022(1)	0.0081(4)	-0.0008(4)	0.0043(7)	0.0006(4)
O(2)	0.1705(4)	0.0614(2)	0.2083(4)	0.0093(5)	0.0026(1)	0.0098(5)	-0.0014(4)	-0.0006(8)	-0.0012(4)
O(3)	0.0501(4)	0.1675(2)	0.0146(4)	0.0124(5)	0.0020(1)	0.0109(5)	-0.0016(4)	0.0059(8)	-0.0007(4)
O(4)	-0.1610(4)	0.0321(2)	0.1019(5)	0.0098(5)	0.0027(1)	0.0151(6)	0.0018(5)	0.0100(8)	0.0005(5)
C(1)	0.1153(7)	0.0677(3)	-0.2279(7)	0.0121(7)	0.0029(2)	0.0105(7)	-0.0001(7)	0.0111(1)	0.0026(6)
C(2)	0.0510(8)	0.1349(4)	-0.3089(8)	0.0174(10)	0.0037(3)	0.0134(9)	0.0014(9)	0.0009(1)	0.0060(8)
C(3)	0.2653(7)	0.0834(4)	-0.1423(6)	0.0091(7)	0.0046(3)	0.0176(10)	-0.0026(8)	0.0101(1)	0.0007(9)
C(4)	0.2491(7)	0.0941(4)	0.2916(6)	0.0097(7)	0.0034(2)	0.0099(7)	-0.0003(7)	-0.0031(1)	0.0016(8)
C(5)	0.2331(9)	0.0134(5)	0.4438(7)	0.0185(12)	0.0066(4)	0.0108(9)	0.0005(12)	0.003(2)	0.0029(11)
C(6)	0.4001(8)	0.0152(5)	0.2799(9)	0.0097(8)	0.0045(3)	0.0210(11)	0.0004(9)	0.002(2)	0.0015(11)
C(7)	0.0972(8)	0.2232(4)	0.1190(8)	0.0186(11)	0.0021(2)	0.0161(10)	-0.0038(8)	0.006(2)	-0.0023(8)
C(8)	0.0268(10)	0.2970(5)	0.0607(10)	0.0278(17)	0.0024(3)	0.0264(17)	0.0003(11)	0.011(3)	-0.0018(10)
C(9)	0.2545(9)	0.2317(5)	0.1447(11)	0.0165(11)	0.0047(3)	0.0260(15)	-0.0007(10)	-0.001(2)	-0.0024(12)
C(10)	-0.2292(7)	0.1526(4)	0.1115(8)	0.0123(7)	0.0029(2)	0.0166(9)	0.0055(7)	0.009(1)	-0.0001(8)
C(11)	-0.3765(10)	0.1467(6)	0.0167(13)	0.0193(13)	0.0062(4)	0.0329(20)	0.0107(12)	-0.013(3)	-0.0038(16)
C(12)	-0.2447(12)	0.1630(6)	0.2615(10)	0.0460(19)	0.0076(4)	0.0205(13)	0.0232(13)	0.027(2)	0.0019(13)

The form of the anisotropic thermal parameter is: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

Table IV. POSITIONAL AND THERMAL PARAMETERS AND THEIR ESTIMATED STANDARD DEVIATIONS, for $\text{Mo}_2(\text{OP}^1)_6(\text{NO})_2$.

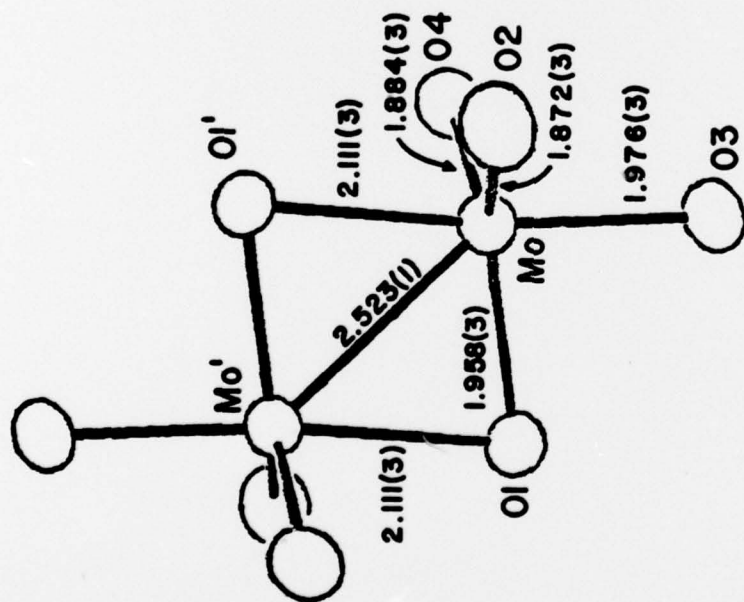
Atom	X	Y	Z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Mo(1)	-0.1085(1)	0.05358(8)	0.0592(1)	0.0133(1)	0.00887(6)	0.0181(1)	-0.0010(1)	0.0164(2)	0.0008(2)
Mo(2)	0.3931(1)	0.55137(8)	0.5638(1)	0.0140(1)	0.00647(6)	0.0197(2)	-0.0028(2)	0.0138(2)	-0.0004(2)
O(1)	0.0053(7)	-0.0572(5)	0.0883(6)	0.0133(8)	0.0058(4)	0.018(1)	-0.001(1)	0.017(1)	0.004(1)
O(2)	-0.0235(9)	0.1478(6)	0.1392(9)	0.0199(11)	0.0075(5)	0.020(1)	-0.003(1)	0.019(2)	-0.004(1)
O(3)	-0.2702(8)	0.0760(6)	-0.1203(9)	0.0124(9)	0.0088(5)	0.022(1)	-0.004(1)	0.012(2)	0.002(1)
O(4)	-0.2279(10)	0.0250(8)	0.2695(11)	0.0302(13)	0.0154(9)	0.036(1)	-0.001(2)	0.049(2)	0.007(2)
O(5)	0.4039(7)	0.5251(5)	0.3764(8)	0.0094(9)	0.0085(5)	0.015(1)	-0.001(1)	0.003(2)	0.001(1)
O(6)	0.4957(10)	0.6310(6)	0.6820(11)	0.0242(14)	0.0083(6)	0.027(2)	-0.006(1)	0.020(2)	-0.006(2)
O(7)	0.3676(8)	0.4652(6)	0.6717(9)	0.0164(10)	0.0080(5)	0.025(1)	-0.005(1)	0.020(2)	0.000(1)
O(8)	0.1159(11)	0.6538(7)	0.4427(13)	0.0214(14)	0.0106(7)	0.040(2)	0.009(2)	0.024(3)	0.003(2)
N(1)	-0.181(1)	0.0355(8)	0.182(1)	0.021(1)	0.0113(8)	0.024(1)	0.004(2)	0.030(2)	0.004(2)
N(2)	0.226(1)	0.6108(7)	0.489(1)	0.017(1)	0.0085(7)	0.026(2)	0.000(2)	0.021(2)	0.002(2)
C(1)	0.034(1)	-0.1349(9)	0.182(2)	7.7(4)					
C(2)	0.109(2)	-0.1130(11)	0.357(2)	9.6(5)					
C(3)	-0.103(2)	-0.1690(11)	0.139(2)	9.4(5)					
C(4)	-0.011(2)	0.1915(11)	0.274(2)	9.3(5)					
C(5)	0.121(2)	0.1494(14)	0.416(2)	13.4(7)					
C(6)	-0.103(2)	0.2814(14)	0.250(2)	13.9(7)					
C(7)	-0.413(2)	0.0696(11)	0.140(2)	10.3(5)					
C(8)	-0.19(2)	0.1504(16)	-0.212(3)	16.2(8)					
C(9)	-0.470(3)	0.0096(17)	-0.200(3)	17.7(10)					
C(10)	0.304(2)	0.5507(10)	0.215(2)	8.0(4)					
C(11)	0.175(2)	0.5100(13)	0.173(2)	12.4(6)					
C(12)	0.299(2)	0.6457(13)	0.196(2)	12.0(6)					
C(13)	0.451(2)	0.7252(14)	0.698(2)	13.6(7)					
C(14)	0.492(3)	0.7677(18)	0.596(3)	18.9(10)					
C(15)	0.474(4)	0.7357(22)	0.859(4)	23.8(13)					

Table IV. (continued)

C(16)	0.239(2)	0.4529(11)	0.686(2)	19.4(5)
C(17)	0.237(3)	0.3565(17)	0.682(3)	16.8(9)
C(18)	0.245(2)	0.4928(16)	0.825(3)	15.6(8)

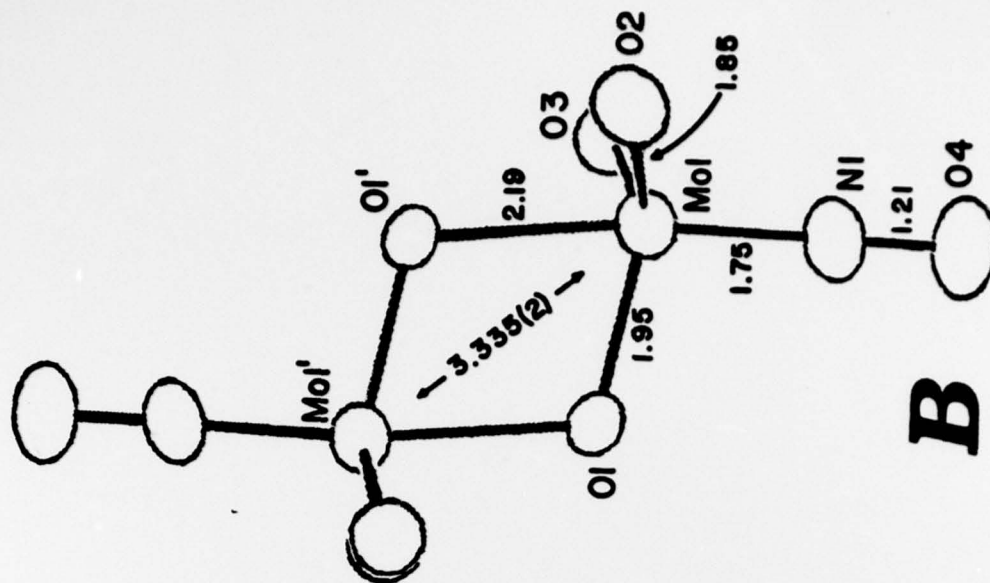
The form of the anisotropic thermal parameter is: $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

Figure 1. Coordination geometries of (A) $\text{Mo}_2(\text{OPr}^1)_8$ and (B) $\text{Mo}_2(\text{OPr}^1)_6(\text{NO})_2$ showing some pertinent bond distances. Distances shown for b are averaged over two independent molecules. In both A and B the molecules possess rigorous C_1 and virtual C_{2v} symmetry.



A

$\text{Mo}_2(\text{O-i-Pr})_8$
Skeleton



B

$\text{Mo}_2(\text{O-i-Pr})_6(\text{NO})_2$
Skeleton

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